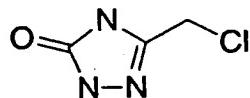


WHAT IS CLAIMED IS:

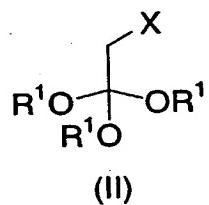
1. A process for the preparation of 3-chloromethyl-1,2,4-triazolin-5-one, of formula (I):



(I)

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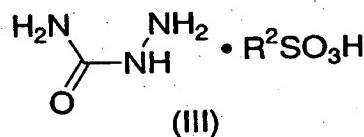
- comprising reacting a triaryl- or trialkylorthoester of formula (II):



(II)

wherein X is a halide, and each R¹ independently is a C₁₋₁₀alkyl or aryl, with a sulfonic acid salt of semicarbazide of formula (III):

10



(III)

wherein R² is a C₁₋₁₀alkyl, C₁₋₁₀alkylhalo, C₅₋₁₆cycloalkyl, or aryl, in an organic solvent, and collecting the resultant compound of formula (I).

- 15 2. The process of Claim 1 wherein R² of the sulfonic acid salt of formula (III) is methyl.
3. The process of Claim 1 wherein R² of the sulfonic acid salt of formula (III) is trifluoromethyl.
- 20 4. The process of Claim 1 wherein R² of the sulfonic acid salt of formula (III) is camphor-10-yl.
5. The process of Claim 1 wherein R² of the sulfonic acid salt of formula (III) is para-tolyl.

6. The process of Claim 1 wherein the organic solvent comprises an alkyl alcohol.

5 7. The process of Claim 6 wherein the alkyl alcohol is methanol.

8. The process of Claim 1 wherein X is chloride.

9. The process of Claim 1 wherein each R₁ of formula (II) is methyl.

10 10. The process of Claim 1 wherein each R₁ of formula (II) is phenyl.

11. The process of Claim 1 wherein the reaction temperature is maintained at about 20-70°C.

15 12. The process of Claim 1 wherein the reaction temperature is maintained at about 35-45°C.

20 13. The process of Claim 1 wherein the reaction mixture is maintained for about 1-24 hours before collecting the compound of formula (I).

14. The process of Claim 1 wherein the reaction mixture is maintained for about 5-20 hours before collecting the compound of formula (I).

25 15. The process of Claim 1 wherein the reaction mixture is maintained for about 10-16 hours before collecting the compound of formula (I).

16. The process of Claim 1 wherein the collection of the compound of formula (I) comprises the steps of:

30 concentrating the reaction mixture,

adding an organic solvent to the concentrated reaction mixture,

cooling the mixture to result in the formation of a solid product,

isolation of the solid product,

washing the solid product with an ethereal solvent,

35 contacting the washed solid product with an aqueous acid for about 1-6 hours,

and isolation of the compound of formula (I).

17. The process of Claim 16, wherein the organic solvent is immiscible with
water.

5 18. The process of Claim 17, wherein the organic solvent is methyl *t*-butyl
ether.

10 19. The process of Claim 17, wherein the organic solvent is toluene.

10 20. The process of Claim 16, wherein the aqueous acid comprises about 0.5-5
N hydrochloric acid.

15 21. The process of Claim 16, wherein the aqueous acid comprises about 0.5 to
5 N trifluoroacetic acid.

20 22. The process of Claim 1 wherein the collection of the compound of formula
(I) comprises the steps of:

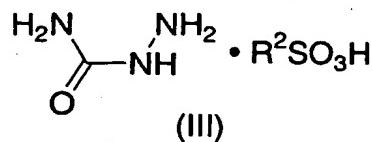
concentrating the reaction mixture,
adding a brine solution to the concentrated reaction mixture,
cooling the mixture to result in the formation of a solid product,
isolation of the solid product,
contacting the solid product with an aqueous acid for about 1-6 hours,
and isolation of the compound of formula (I).

25 23. The process of Claim 22, wherein the brine solution comprises an aqueous
sodium chloride solution.

30 24. The process of Claim 22, wherein the aqueous acid comprises about 0.5 to
5 N hydrochloric acid.

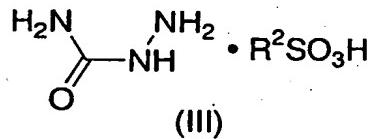
35 25. The process of Claim 22, wherein the aqueous acid comprises about 0.5 to
5 N trifluoroacetic acid.

35 26. A substantially pure sulfonic acid salt of semicarbazide of formula (III):



wherein R² is a C₁₋₁₀alkyl, C₁₋₁₀alkylhalo, C₅₋₁₆cycloalkyl, or aryl.

- 5 27. The sulfonic acid salt of formula (III) of Claim 26 wherein R² is methyl.
28. The sulfonic acid salt of formula (III) of Claim 26 wherein R² is trifluoromethyl.
- 10 29. The sulfonic acid salt of formula (III) of Claim 26 wherein R² is camphor-10-yl.
30. The sulfonic acid salt of formula (III) of Claim 26 wherein R² is *para*-tolyl.
- 15 31. A process for the preparation of a sulfonic acid salt of semicarbazide (III):



wherein R² is a C₁₋₁₀alkyl, C₁₋₁₀alkylhalo, C₅₋₁₆cycloalkyl, or aryl, comprising the steps of:

20 adding a solution of ammonia in a first alcoholic solvent to a slurry of semicarbazide hydrochloride suspended in a second alcoholic solvent,

 filtering off the resulting ammonium chloride,

 diluting the filtrate with an organic solvent,

 treating the solution so obtained with a slight molar excess of an alkyl or aryl

25 sulfonic acid,

 and collecting the sulfonic acid salt of semicarbazide (III).

32. The process of Claim 31 wherein the first and the second alcoholic solvents are methanol.
- 5 33. The process of Claim 31 wherein the organic solvent is immiscible with water.
- 10 34. The process of Claim 33 wherein the organic solvent is ethyl acetate.
35. The process of Claim 33 wherein the organic solvent is methylene chloride.
- 15 36. The process of Claim 33 wherein the organic solvent is methyl-*t*-butyl ether.
37. The process of Claim 33 wherein the organic solvent is toluene.
- 20 38. The process of Claim 31 wherein R² of the sulfonic acid salt of formula (III) is methyl.
39. The process of Claim 31 wherein R² of the sulfonic acid salt of formula (III) is trifluoromethyl.
- 25 40. The process of Claim 31 wherein R² of the sulfonic acid salt of formula (III) is camphor-10-yl.
41. The process of Claim 31 wherein R² of the sulfonic acid salt of formula (III) is *para*-tolyl.